“Synthesis and Electrochemical Properties of Heterojunction Nanostructures”

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1) Introduction

With the ever-increasing adaption of large-scale energy storage systems and electric devices, the energy storage capability of batteries and supercapacitors has faced increased demand and challenges. The electrodes of these devices have experienced radical change with the introduction of nano-scale materials. Novel materials consistings have attracted increasing attention due to their unique interfaces, robust architectures, and synergistic effect, and thus, ability to enhance the energy/power outputs as well as the lifespan of batteries [1].

In the past decades, energy storage devices have drawn widespread attention in diverse fields including hybrid electric vehicles and smart portable electronics. By taking advantage of two different electrode materials, asymmetric supercapacitors (ASCs) can extend their operating voltage window and display more excellent energy-storage capacity than those of symmetric supercapacitors. Recent developments have demonstrated that the transition metal sulphides (TMSs) have emerged as a promising class of materials for ASCs due to their low-cost, fascinating redox reversibility and electronic conductivity [2].
Heterojunction is the interface between any two solid-materials, including crystalline and amorphous structures of metallic, insulating, fast ion conductor and semiconducting. Heterojunctions are promising structures to induce novel physical phenomena and useful functionality from fundamental science to engineering applications.

The heterojunction can provide approach to investigate the fundamental properties and the physical origins systematically. On the other hand, the findings from the investigation of the heterojunction will open the door to create novel devices and develop new academic fields. Facilitating discovery of interlayer exchange coupling oscillation as a function of space layer thickness. These physical phenomena are derived from spin-dependent scattering and interferences. The control and manipulation of interlayer exchange coupling lead to the development of synthetic antiferromagnetic structures. The heterojunction systems also serve as a guideline for the development of more complex nanosystems with optimized properties [3].

Composition can be efficient for increasing the dopant solubility, increasing the activation rate by lowering the ionization energy of acceptors and donors, and increasing the carrier mobility. Enhanced dopant incorporation is especially significant under no equilibrium growth conditions owing to the introduction of new chemical species as co-dopants.

Electrochemical Hydrogen Evolution

The development of sustainable clean energy captures great attention of scientists in recent years due to the ever-increasing environmental pollution and energy requirements. Molecular Hydrogen (H_2) is considered as a promising candidate to meet global terawatt energy requirement in the future. Three principle steps typically occur to convert H^+ to H_2 and to realize the hydrogen evolution reaction (HER), ($2H^+ + 2e^- \rightarrow H_2$) process in acidic media that including the adsorption of protons, electrochemical reduction, and desorption of hydrogen gas [4,5].

Therefore, efficient and durable electrocatalysts are usually required to minimize the overpotential and to reduce the free energy barriers (i.e., ΔGH) of adsorbed hydrogen species, which make the HER process more prone to proceed efficiently in both thermodynamics and kinetics.

Catalysis

Catalysis is the process of increasing the rate of a chemical reaction by adding a substance known as a catalyst which is not consumed in the catalyzed reaction and can continue to act repeatedly. Because of this only very small amounts of catalyst are required to alter the reaction rate in principle [6]. The production of most industrially important chemicals involves catalysis. Similarly, most biochemically significant processes are catalyzed. Research into catalysis is a major field in applied science and involves many areas of chemistry and physics, notably organometallic chemistry and the materials science. Catalysis is relevant to many aspects of environmental science [7].

In general, chemical reactions occur faster in the presence of a catalyst because the catalyst provides an alternative reaction pathway with lower activation energy than the non-catalyzed mechanism. In catalyzed mechanisms, the catalyst usually reacts to form a temporary intermediate, which regenerates the original catalyst in a cyclic process. A substance which provides a mechanism with higher activation energy does not decrease the rate because the reaction can still occur by the non-catalyzed route. The added substance which does reduce the reaction rate is not considered a catalyst but a reaction inhibitor. Catalysts may be classified as either homogeneous or heterogeneous. A homogeneous catalyst is one whose molecules are dispersed in the same phase (usually gaseous or liquid) as the reactant's molecules. A heterogeneous catalyst is one whose molecules are not in the same phase as the

reactants, which are typically gases or liquids that are adsorbed in to the surface of the solid catalyst. Enzyme e.g. and other biocatalyst e.g. are often considered as a third category [8].

2)-Literature review

Hou et al. 2014 their study reported that SnO_2 & TiO_2 heterojunction nanostructures for lithium-ion batteries and self-powered UV photodetectors with improved performances.

An efficient technique for creating a hybrid material comprising of rutile SnO_2 nanoparticles and rutile TiO_2 nanorods is investigated in this work. After 100 discharge/charge cycles at 200 mAg^{-1}, the as-prepared SnO_2 & TiO_2 material, directly produced on a carbon cloth as a binder-free anode, has a reversible capacity of 700 mAh g^{-1}. A high photocurrent response of 0.1 mA cm^2, as well as an exceptional self-powered and rapid response and "visible blind" properties, were reported. A hybrid material like this might have a complementing impact in lithium-ion batteries and a better band gap match in photovoltaic devices, and could therefore be used to dye-sensitized solar cells and supercapacitors [9].

Xiao et al. 2021 reported photoelectrochemical properties of TiO_2/Sb_2S_3 heterojunction with CuO nanostructure as photocathode. When charge transfer rate in device with CuO seed layer photocathode is significantly enhanced. The better hole transfers also improved the charge separation in Sb2S3 and reduced their recombination rate, resulting in the enhanced photocurrent and photovoltage by CuO nanocones array photocathode [10].
Adimule et al. 2021 reported synthesis of hierarchical structured Gd-doped α-Sb_2O_4 as an advanced nanomaterial for high performance energy storage devices.

Bimetallic oxide nanostructures of Gdx; Sb_2O_4 (x = 5, 8, 10 wt. percent) appeared as a novel electrode material for batteries, according to this study, since they have a high specific capacity and cyclic stability. They used a co-precipitation technique for synthesising Gdx (x = 5, 8, and 10% by weight): Sb_2O_4 with excellent electrochemical characteristics. XRD, SEM, UV-Visible, BET, energy dispersive spectroscopy, and XPS spectroscopic examinations were used to evaluate Gd; Sb_2O_4 produced by co precipitation. The specific capacitance of Gd; Sb_2O_4 (10 wt. percent) in 6 M KOH aqueous solution was 958 mAh/g at a current density of 0.15 A/g, with a capacitance retention rate of 86.5 percent after 1000 cycles. They presented a simple approach for synthesising Gdx; Sb_2O_4 NS, which are promising electrode materials for high-performance super capacitor applications [11].

Dai *et al.* reported hierarchical electrode architectures for high energy lithium-chalcogen rechargeable batteries. In this study they synthesized of SiO_2 obtained by filtration and dried under vacuum before using. Synthesis of the material is based on a modified reduction method. Also, they did material characterization by the X-ray powder diffractometer. The size and morphology of products were determined by scanning electron microscopy and transmutation electron microscopy. As well they did electrochemical evaluation cyclic-voltammetry measurements were performed using a bio-logic. They had developed hierarchical electrode architectures (SPC) by a wet synthesis method for Li-chalcogen battery applications. Excellent electrochemical result was obtained for both Li-S and Li-Se without external additives or modifications of electrode or cell configuration [6].

Arora *et al*. studied nanostructure $moS_2/BivO_4$ composites for energy storage applications**.**

They reported optimal synthesis and electrochemical characterization of a low-layer nanostructured moS_2 compound along with $BiVO_4$. When compared to pure $BiVO_4$ and a graphene/$BiVO_4$ combination, the $moS_2/BiVO_4$ nanocomposite offers superior freight storage, longer discharge periods, and greater stability. Small moS_2 molecules with a clearly dimensioned lateral dimension give a noticeable capacitive component, which aids in raising the specific amplitude, according to the findings. They also described how to use an aqueous electrolyte to get the best mix of single-layer $BiVO_4$ and low-layer nanoparticles MoS_2.

The results reveal that the moS_2/ $BiVO_4$ nanocomposite will be a good contender for high-energy storage applications [12].

Jin W et al. studies recent advances of porous transition metal-based nonmaterials for electrochemical energy conversion and storage applications. In this studies discussed the recent achievements in porous transition metal-based nonmaterials for electrochemical energy storage and conversion, including conventional oxides, sulphide, phosphide, and carbides. They have also, done investigated the current obstacles and future possibilities of porous transition metal-based nonmaterials in electrochemical energy storage and conversion applications, as well as the designed, manufacturing, and application performance of nonmaterials are illustrated and compared [13].

Sakthivel *et al*. reported synthesis and characterization of bimetallic nickel-cobalt chalcogenides ($NiCoSe_2$, $NiCo_2S_4$, and $NiCo_2O_4$) for non-enzymatic hydrogen peroxide sensor and energy storage: Electrochemical properties dependence on the metal-to-chalcogen

composition. In this work, they employed a simple hydrothermal approach to show the synthesis of $NiCoSe_2$, $NiCo_2S_4$, and $NiCo_2O_4$ with various architectures, using electrode materials for electrochemical sensor and super capacitor applications. As findings clearly show edsuperior $NiCoSe_2$ activity of than $NiCo_2O_4$ and $NiCo_2S_4$ in the electrochemical detection of H_2O_2. H_2O_2 was detected with an ultra-low detection limit (0.03 mM) and sensitivity (8.46 mA mM^{-1} cm^{-2}) using the $NiCoSe_2$ modified electrode. Finally, they discovered that $NiCoSe_2$ is an excellent electrode material for electrochemical sensors and supercapacitors. Design and fabrication of ZnO/CdS heterostructured nanocomposites for enhanced hydrogen evolution from solar water splitting [5].

Revathi, Saravanan, and others 2021 designed and fabricated of ZnO/CdS heterostructured nanocomposites for enhanced hydrogen evolution from solar water splitting.

In this report, a ZnO/CdS nanosheets / nanoparticles hetero-structure was created using a simple and one-step microwave irradiation approach. The phase formation of ZnO and CdS in ZnO/CdS nanocomposites through XRD and Raman spectroscopy. The optical bandgap energy (Eg) was calculated to be between 3.13 and 2.29 eV. Two emissions were seen in the PL spectra at 385 nm and 510 nm, which might be attributed to CdS band edge emission or oxygen vacancies in ZnO. The H_2 photocatalytic experiment was carried out in the presence of sun light. The inclusion of CdS in the host matrix ZnO was shown to be crucial in the creation of ZnO/CdS heterostructures with improved interfaces for direct Z-scheme processes with faster hydrogen evolution rates [14].

Hou and Chen 2021 reported one-step synthesis CdS/single crystal ZnO nanorod heterostructures with high photocatalytic H2 production ability. CdS/single crystal ZnO nanorod heterostructure (CdS/ZnO) was fabricated via microwave synthesis method, and the structural, morphological and photocatalytic hydrogen generation was tested. Studied results demonstrated that CdS particles were decorated on the surface of single crystal ZnO nanorod with crystallite size about 43 nm, and CdS nanoparticle significantly enhanced the photocatalytic hydrogen generation ability of the heterostructure by increasing the specific surface area, promoting the separation, migration of photoinduced carriers and increasing the light absorption. Pure ZnO exhibited bandgap energy of 3.09 eV, while the CdS/ZnO sample possessed a more narrow band gap (2.62 eV) than ZnO nanorod. Under the irradiation of visible light, the highest photocatalytic hydrogen production rate of 4076 μmolg−1h−1 was noted over the CdS/ZnO with the content of CdS is 38 wt. %, which is about 13.59 and 76.91

times more active than pure ZnO nanorod and CdS nanoparticle. In addition, the specific surface area of the composite sample reaches 43.375 m2/g, and the instantaneous photocurrent reaches 130 μA /cm2. The present microwave radiation method was simple, easy to control, and timesaving, thereby providing a efficient route for the preparation of metal oxide-based photocatalytic material with high hydrogen evolution activity [15].

Sharma and Basu 2021 reported Fabrication of centimetre-sized Sb_2S_3/SiO_2 monolithic mimosa pudica nanoflowers for remediation of hazardous pollutants from industrial wastewater. Here, 'mimosa pudica' flower-like Sb_2S_3/SiO_2 monoliths were prepared by impregnation of antimony chloride solution in silica monoliths followed by treatment with potassium thiocyanate, refluxing and calcination. The nanoflower-like morphology of the synthesized monoliths was verified using field emission scanning electron microscope images. The purity/crystallinity and the elemental state of the sulphide were confirmed using X-ray diffraction analysis and X-ray photon spectroscopy whereas energy dispersive spectroscopy verified the homogeneous elemental distribution of the catalyst. The diffuse reflectance spectroscopy and photoluminescence spectroscopy confirmed that Sb_2S_3/SiO_2 monoliths had a narrow band gap (~1.57 eV) and low recombination rate of photoinduced charge carriers. Thermal stability of the materials was also investigated by thermogravimetric analysis [16].

Zhou H et al. reported acetylacetone-directed controllable synthesis of Bi_2S_3 nanostructures with tuneable morphology. In this report, bi_2S_3 microscopic and solve thermally micro flowers in the acetylasone solution were uniformly synthesized by pyrolysis of the Bi_2S_3 diethysone complex without any molds or surfactants. Bi_2S_3 microscopy was manufactured consisting of nanorods with a diameter of 20-40 nm at 180 °C for 12 h . In similar conditions at 240°C for 3 days, micro flowers consisting of nanowires were obtained with lengths of up to several micrometers and a diameter of 20-40 nm. Here electrochemical experiments used by Bi_2S_3 in a lithium ion battery indicated that the first discharge capacity of Bi_2S_3 microphones could reach about 148 mA/h [17].

Shaheen et al. 2021 Synthesised a facile ZnO: NiO-PdO-Pd nanomaterial by organic fuel: Environmentally benign electrode material for energy storage using hydrothermal route. The greener organic synthesis fuel extracted from leaves of Euphorbia cognata Boiss. The obvious Nanospherical particles with crystal size of $21\,nm \pm 2$ were obtained. The organic assisted ZnO: NiO-PdO-Pd nanocomposite was integrated into electrode utilizing substrate in

foam of nickel applying the thermal vacuum treatment. The supercapacitive energy storage potential was first investigated by cyclicvoltammetry by scanning the electrodes at varies rates ranging 2 mV/s to 300 mV/s. By CV ZnO: NiO-PdO-Pd electrode exposed maximum specific capacitance, that is, 348 F/g. Then, galvanostatic charge-discharge analysis was conducted for prepared electrode applying different current densities ranging 0.5 to 30 A/g. Greater energy density, that is, ~10 W/kg was obtained for ZnO: NiO-PdO-Pd electrode having enhanced power density, that is, 4965.6 W/Kg from GCD measurement. Finally, the electrochemical conductivity was investigated by electrochemical impedance spectroscopy. Internal resistance observed in this work was very less, that is, 0.49 Ω, signifying good conductivity because of fast diffusion shown by electrons as well as ions. Consequently, good redox supercapacitors behaviour of ZnO: NiO-PdO-Pd was unveiled, has proven substantial potential of ZnO: NiO-PdO-Pd electrode for energy storage. They have presented a comprehensive approach on phyto fuelled doped ternary mixed MOs ZnO: NiO-PdO-Pd nanomaterial preparation, reorganization of phyto-stabilizing agents as well as reducing agents in attained nanocomposite exploring its electrochemical behaviour for supercapacitor for the very first time [18].

Nagappagari et al. 2021 studied Enhanced photoelectrochemical activity using $NiCo_2S_4$ / spaced TiO_2 nanorod heterojunction. In this study, a well-aligned spaced TiO_2 nanorod arrays were uniformly grown on a fluorine-doped tin oxide substrate, and thermal exfoliation of TiO_2 nanorod, which appeared as small islands, was investigated. The calcination time, which ranged from 1 to 5 h at 400 °C, had a significant impact on the agglomerated TiO_2 nanorod islands, which tended to be uniformly exfoliated as spaced nanorods and eventually improved the PEC performance.

the heterojunction formation with $NiCo_2S_4$ via the successive ionic layer adsorption and reaction (SILAR) method, substantially improve PEC performance, owing to reduced charge carrier recombination at the interface of the heterojunction. they succeeded in improving the photoelectrochemical performance of TiO_2 by heterojunction construction with $NiCo_2S_4$ nanolayers, as well as narrow optimization of exceptional parameters [19].

Iqbal et al. 2021 objective performance supercapacitor based on MoS_2 & TiO_2 composite for wide range temperature application. They used a hydrothermal technique to make a MoS_2 & TiO_2 composite with a flower-like shape. At 10 mV/s, the specific capacitance of MoS_2, MoS_2 & 15 % TiO_2, and MoS_2 & 30 % TiO_2 is determined to be 120, 210, and 126 F/g,

respectively. Furthermore, when the working temperature of the MoS_2 & 15 % TiO_2 based supercapacitor is increased from 25 to 60 °C, the specific capacitance value increases by 200 percent. The higher performance of MoS_2 & 15 % TiO_2 composite over MoS_2 shows that TiO_2 nanorods can be incorporated into MoS_2 and that it could be a good candidate for supercapacitor applications. At room temperature, MoS_2 & 15 % TiO_2 has the highest specific capacitance of 210 F/g at 10 mV/s, as well as outstanding cycling stability (98 percent, 2000 cycles). The results of the impedance spectroscopic investigation show a considerable reduction in interfacial impedances, resulting in a superior capacitance effect combined with favourable electrolytic charge dynamics. The energy density with the maximum power density is stated to be 21 Wh/kg [20].

Wang et al. 2021 synthesised MoS_2 meets TiO_2: facile synthesis strategies, hybrid nanostructures, synergistic properties, and photocatalytic applications. In this paper, TiO_2 /MoS_2 hybrid materials are discussed, as well as the production procedures for the hybrid system's photocatalytic performance. The catalytic synergistic effects of molybdenum disulphide (MoS_2) and titanium dioxide (TiO_2) nanocomposites with outstanding photocatalytic activity were established. This improves the performance of TiO_2 / MoS_2 - based nanocomposites, broadening its potential applications in photocatalysis and rechargeable lithium/sodium-ion batteries. Hydrothermal methods, atomic layer deposition methods, and mechanochemical synthesis methods are all detailed descriptions of common TiO_2 / MoS_2 hybrid material fabrication processes. The mechanisms of photocatalyst enhancement [21].

2) Objectives of the study

The research work will be focus on the following objectives: -

(1) Synthesize and characterize metal oxides/sulphide heterojunction electrode materials.
(2) Synthesize and characterization of heterojunction based
(3) Study of Structure characterization of metal oxide and sulphide compounds by using XRD, SEM, TEM and Raman spectrometer techniques.
(4) Investigate the influence of heterojunction on the supercapacitor and electrochemical HER performances.
(5) Study the supercapacitor performance of the fabricated heterojunction electrodes.
(6) Study performance of the prepared electrodes in electrocatalytic (HER) and (OER).
(7) Study the catalytic activity of the prepared nanostructured electrode materials.

(8) Compare these results with data reported previously & publish manuscript in international journal.

(9) Design thesis accordingly based on the data available.

4) Methods

Hydrothermal method.

It is important branches of inorganic synthesis. Hydrothermal technique is not just confined to the syntheses and growth of conventional and advanced materials but also take a very broad shape, covering several interdisciplinary branches of science and widely applied.

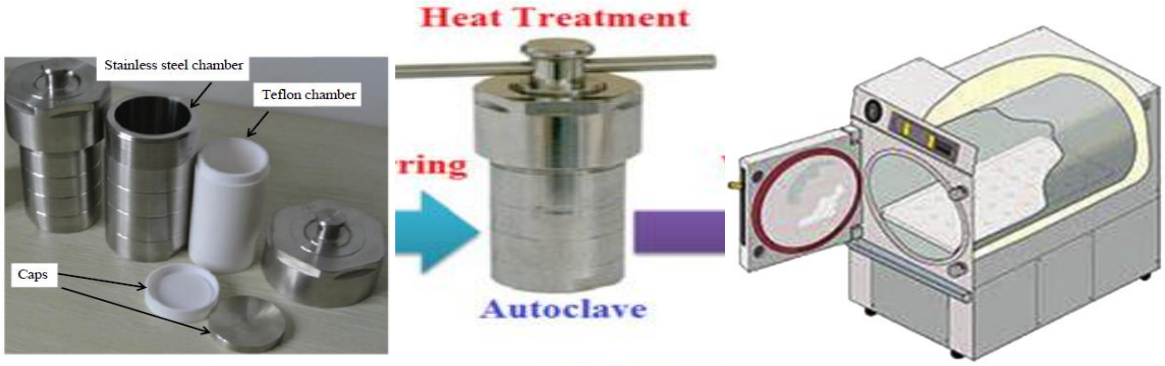

Fig 1: Diagram of hydrothermal method

Hydrothermal method for crystallizing substances from high-temperature aqueous solutions at high vapour pressures; also termed “hydrothermal method.’’ The term “hydrothermal” is of geologic origin. Hydrothermal synthesis can be defined as a method of synthesis of single crystals that depends on the solubility of minerals in hot water under high pressure. The crystal growth is performed in an apparatus consisting of a steel pressure vessel called an autoclave in which a nutrient is supplied along with water. A temperature gradient is maintained between the opposite ends of the growth chamber. At the hotter end the nutrient solute dissolves, while at the cooler end it is deposited on a seed crystal, growing the desired crystal [22].

Chemical bath deposition.

(CBD) is a low-cost, low-temperature method for producing large-area semiconductor thin films (at temperatures ranging from 25 to 90 °C). For the deposition of different nanometal chalcogenide thin films, (CBD) is routinely utilised. The regulated precipitation of the

chemicals from the solution results in excellent deposits on appropriate surfaces. It does not require high-voltage equipment, can operate at room temperature (between 30 and 100 ℃) and is thus affordable. Only an aqueous solution containing a few common chemicals and a substrate for the film to be deposited are required for this deposition method. Transition metal oxide films were created by optimising CBD preparative factors such as deposition temperature, duration, pH, complexing agent, and so on. Various complexing techniques were used to deposit metal oxide films from a solution containing precursor [23].

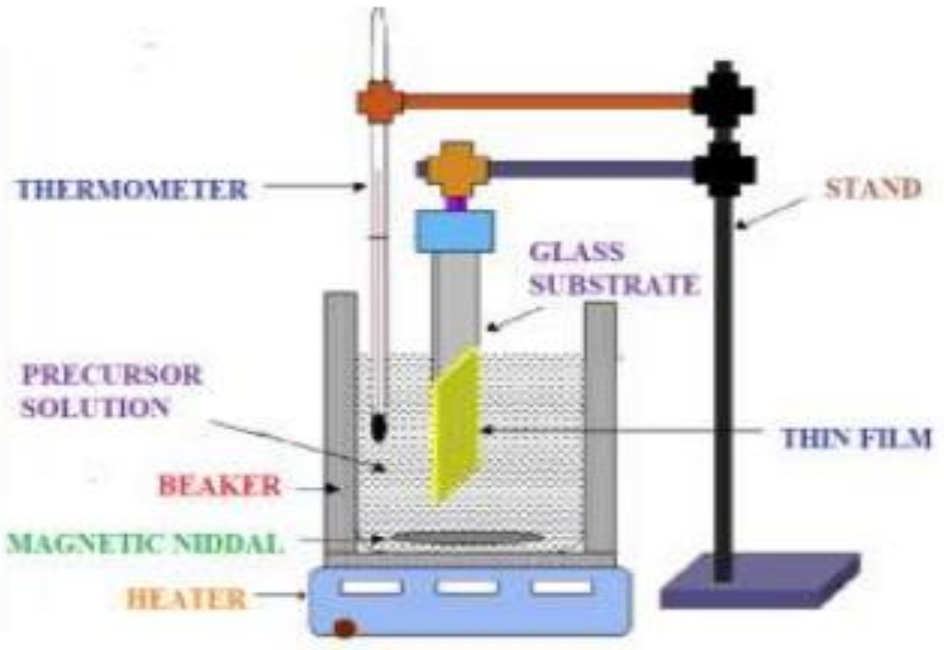

Fig 2: Diagram of chemical bath deposition method

SILAR Method.

The successive ionic layer adsorption and reaction (SILAR) approach is a simple, less expensive, and faster method for the deposition of binary semiconducting thin films. It can also be used for large-area thin film deposition. The process was used to grow a variety of II–VI compounds, including CdS, ZnS, and CdS/ZnS multilayers. The substrate is submerged separately in two precursor solutions and rinsed in between with water to remove loosely bound species in the SILAR process. one SILAR cycle consists of cation precursor adsorption, water rinsing, anion precursor adsorption, reaction, and another rinsing. The thin film's growth, on the other hand, can be precisely regulated to within one SILAR cycle [25].

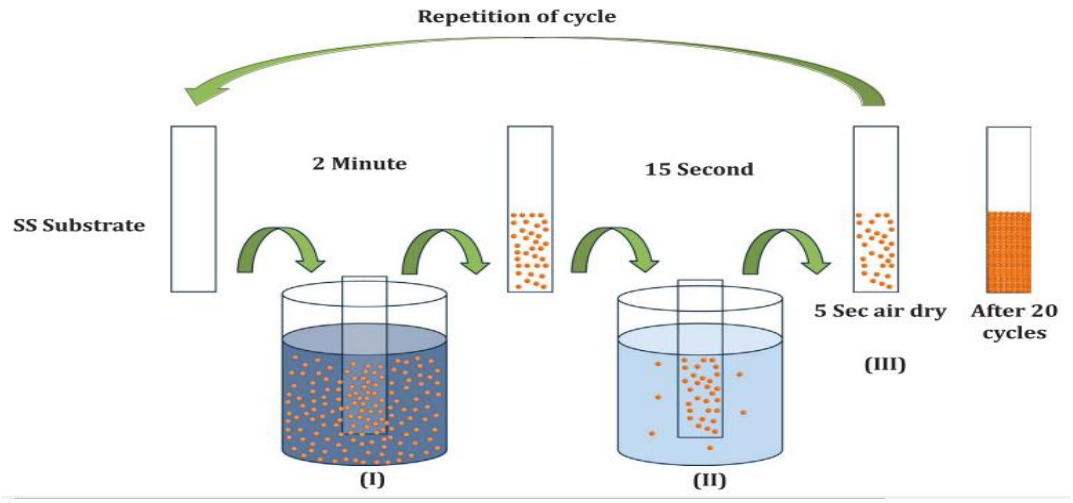

Fig 3: Steps involved in SILAR

Electrodeposition.

Electrodeposition is regarded as one of the most cost-effective ways for fabricating semiconductor thin films. Electrodeposition, often known as electroplating, is a process for depositing material from a solution containing ionic species onto a conducting surface.

This deposition method is most commonly used to deposit thin films of material onto an object's surface to modify its exterior features, such as corrosion resistance, abrasion resistance, ornamental quality, or simply to deposit a layer that is part of a more intricate device. The electrodeposition scheme is depicted in the diagram below. Three electrodes are utilised in this technique: working, reference, and counter (occasionally secondary) electrodes, respectively. The electrodes are attached to a potentiostat, a device that regulates the deposition process. These electrodes were stored in a container with a liquid that contained dissolved ionic species [24].

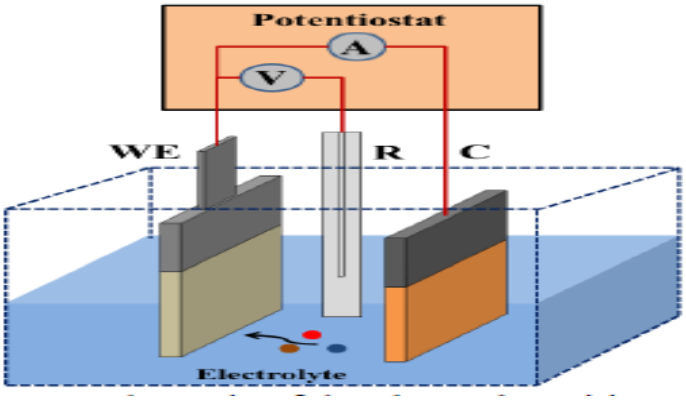

Fig 4: Schedule of Electrodeposition Method

Characterization

Physical characterizations

a) Structural analysis

Diffraction analysis with X-rays (XRD). The diffraction method is also used to distinguish between crystalline and nanocrystalline materials, and this spectroscopic technique is utilised to identify the crystal's atomic and molecular structure. This spectroscopic approach is also used to distinguish the nature of crystalline materials from nanocrystalline materials, and it is used to detect the atomic and molecular structure of crystals.

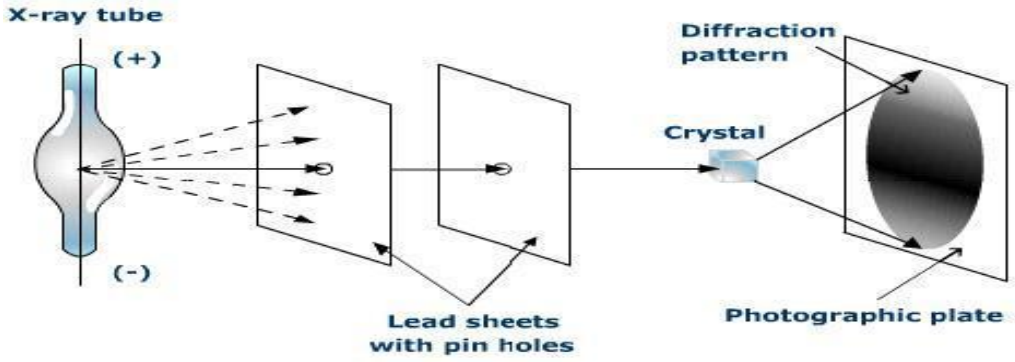

Fig. 5: Ray diagram of X-ray diffractometer

b) Surface morphology study.

SEM is a characterization technique that uses a focused electron beam to scan a solid sample from the optical to nanoscale range. In these types of characterisation processes, secondary electrons are released from the sample and collected to create a secondary emission area map. Field-emission microscopes will be used to determine the external morphology (texture), chemical composition, and crystalline structure of materials.

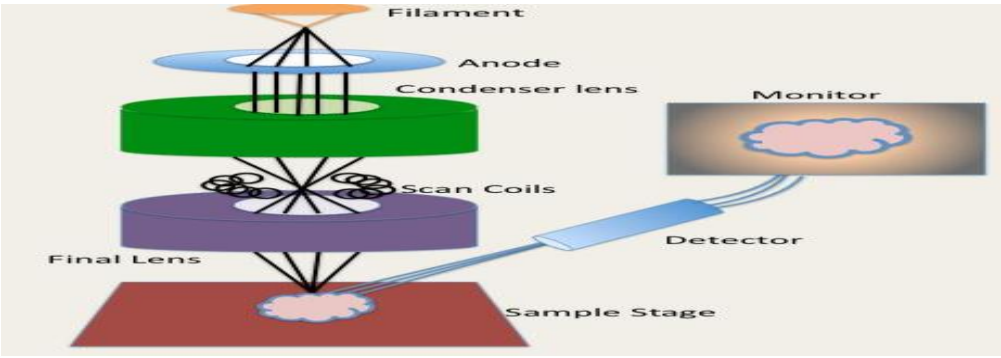

Fig 6 : Scanning electron microscopy

c) **Raman spectrometer**

Raman spectroscopy uses laser light to investigate molecular structure. For every million photons distributed, a single photon will interact with the vibrational states of a sample molecule and emit light of a distinct wavelength. In addition to IR spectroscopy, Raman systems can be used to explore molecular structure.

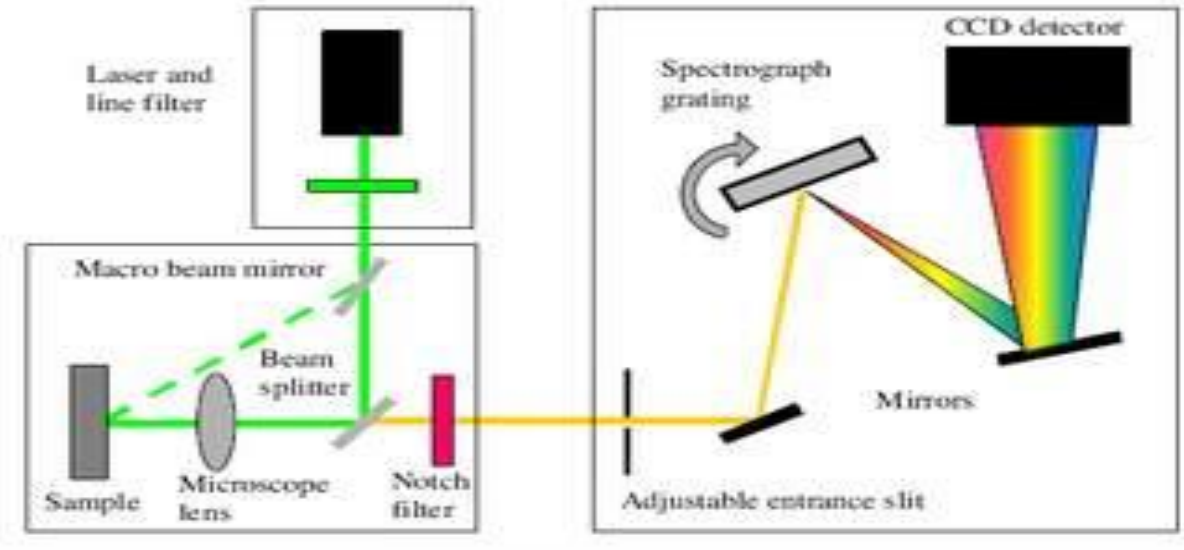

Fig. 7: Schematic diagram of a Raman Spectrometer

d) Measurement with high-resolution electron microscopy

In this method, the electrons are focused directly on the substance. The HR examines the material as a whole, rather than just the surface. It has a magnification range of 50 million and displays a two-dimensional image. An electron beam is fed through an ultrathin material, interacts with it as it passes, and forms an image in a HR with a resolution of 0.5 Angstrom. In both physical and biological sciences, TEM characterizations are used. The high-resolution electron microscope will be used to determine the particle size of nanoparticles. They work in a variety of academic, scientific, and industrial sectors as well.

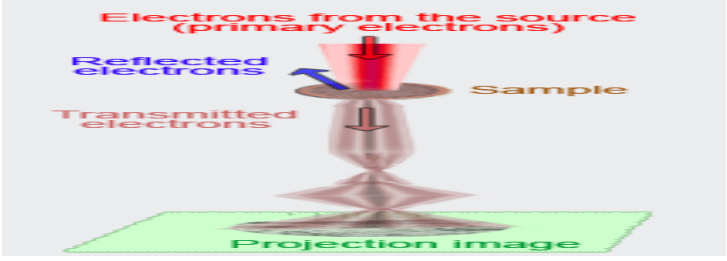

Fig. 8: Schematic diagram of high-resolution electron microscope.

f) X-ray photoelectron spectroscopy (XPS)

Is a surface-sensitive quantitative spectroscopic method that determines the elemental composition, empirical formula, chemical state, and electronic state of the elements present in a material in parts per thousand. Simply said, XPS is a valuable measuring technique since it displays not only what components are included inside a film, but also what other elements they are bound to.

Electrochemical measurement

A three-electrode system on an electrochemical workstation will be used to assess both water spitting and supercapacitor performance. The entire measurement procedure will be carried out in an electrolyte (e.g., 0.5 mL H_2SO_4) with a counter electrode, an Ag/AgCl (in saturated KCl solution) reference electrode, and a catalyst-modified GCE that was generated using the methods above as the working electrode.

a) Cyclic voltammeter.

CV is an electrochemical method for determining the current that occurs in an electrochemical cell when the voltage exceeds that predicted by the Nernst equation. CV may be used to investigate qualitative information about electrochemical processes under a variety of situations, such as the existence of intermediates in oxidation-reduction reactions and a reaction's reversibility. CV may will be used to estimate a system's electron stoichiometry, an analyst's diffusion coefficient, and the formal reduction potential, which can be used as a tool for identification.

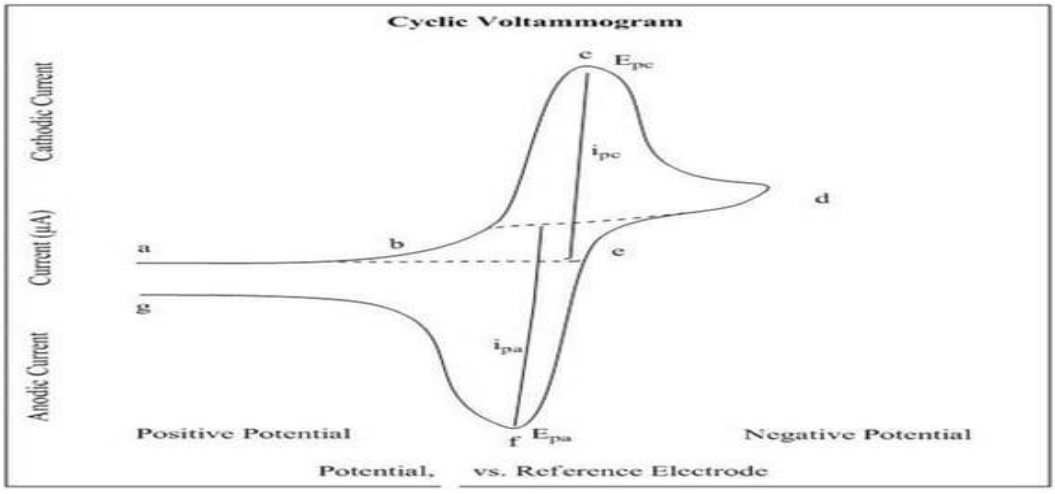

Fig. 9 : Cyclic voltammetry

b) Electrochemical impedance spectroscopy.

ESI is an electrochemical approach for determining the impedance of a system in relation to the frequency of the AC potential. It's also a useful instrument for studying chemical and physical processes in both liquids and solids. (EIS) is a potent nondestructive tool for researching microbial fuel cells that can complement current approaches (MFCs).

c) Galvanostatic charge- discharge.

The charge-discharge curve, is a key metric in supercapacitors that provides all information about the electrode material, such as specific capacitance, stability, power and energy densities, and so on. In a charge-discharge curve with a fixed potential window, the current density remains constant, but it may be modified to get varied discharge time values between the two potentials. The curve in the charge process can start at zero time or any value within the potential window's range and climb linearly to the desired potential. Then it reveres its direction to return to the original value within a certain time frame.

d) Electrochemical hydrogen evolution reaction.

The polarisation curves were created using a variety of sweeping potentials. The j–t curve of chronoamperometry is measured at a constant voltage. In the same scenario, electrochemical impedance spectroscopy is acquired at a certain over potential, frequency range, and amplitude.

e) Oxygen evolution reaction.

Is a limiting reaction in the process of producing molecular oxygen by chemical reactions, such as water oxidation during oxygenic photosynthesis and water electrolysis into oxygen and hydrogen. The success of a variety of renewable energy technologies, including solar fuel generation and metal–air batteries, hinges on the development of better catalysts for the OER. Oxygen is released from water by oxygenic photosynthesis, water electrolysis, and the

thermal decomposition of different oxides.

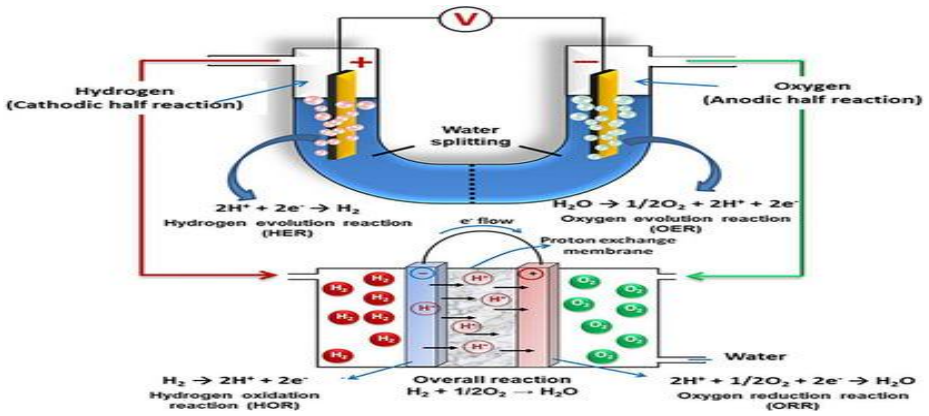

Fig. 11: Operation of water electrolysis unit.

f) Supercapacitor performance

The same material can be used to make electrochemical energy storage devices with two electrodes (an anode and a cathode) of different materials. A piece of nickel foam coated with a particular number of as-synthesized products serves as the working electrode, with a Hg/HgO electrode serving as the reference electrode and a Pt electrode serving as the counter electrode. The use of carbon foam as the negative electrode (negatrode) and a metal oxide as the positive electrode (positrode) in electrochemical asymmetric supercapacitors (ASCs) has recently peaked; however, research into their role in electrocatalysts as the cathode (platinum-carbon) for hydrogen evolution reactions (HERs) and the anode (ruthenium oxide/iridium oxide) for oxygen evolution reactions (OERs) is still Thus, producing two electrodes from the same material with opposing properties is interesting, particularly for materials with potential for use in electrochemical energy storage and water splitting devices. Another is the galvanostatic charge–discharge (GCD) curve [26].

5) Problem undertaken

The wide applications of nanostructured are based on the progress in the synthesis of new nanostructured materials of different sizes and new morphologies. The best materials that can be used as an electrode in store energy, as well as supercapacitor, are Heterojunction materials due to their wide potential window, highly redox reactions, distinct oxidation states, high conductivity, remarkably low over potential, good thermal stability, long cycle life, and high rate capability as well as it is an environment friend. Therefore, further developing new materials with low overpotential and better performance relative to existing electrode

materials is the most important technique to overcome these challenges. And also for the following metal oxides/metal sulphides will be study as:

1) Synthesis of various heterojunction materials
2) Their physical Characterization like structure, composition, surface appearance, optical studies etc.
3) Measurement of supercapacitor properties like CV, CCB, life time, impedance spectroscopy, measuring specific capacitance.
4) Water splitting properties such as OER, HER for measuring overpotential, tafel slope and stability etc.
5) Based on obtained results manuscript in reported international journal will be published

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